ELECTRODEPOSITION OF CARBOXYLATED MULTIWALL CARBON NANOTUBE ON GRAPHITE REINFORCEMENT CARBON FOR VOLTAMMETRY DETECTION OF CADMIUM

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Dedicated to my beloved family…

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ABSTRACT

Determination of cadmium ion at trace and sub-trace levels is still challenging due to high cost and limited capability of analytical instrumentation. A simple, low cost, non-toxic graphite reinforcement carbon (GRC) electrode modified with carboxylated multiwall carbon nanotubes (c-MWCNT) was prepared by electrodeposition process and used for the determination of cadmium ions at sub-part per billion (sub-ppb) levels. The study involved investigation of electrochemical performance of GRC with different hardness and size. The carboxylated-functionalized MWCNT was characterized by Fourier Transform Infrared Spectrophotometer (FTIR) and Field Emission Scanning Electron Microscope-Energy Dispersive X-ray analysis (FESEM-EDX). FESEM was also used to investigate the surface morphology of the c-MWCNT/GRC electrode. The newly developed electrode was successfully used for the detection of cadmium ion in 0.04 M Briton Robinson Buffer (BRB) by differential pulse anodic stripping voltammetry (DPASV). Some important operational parameters including pH of the buffer, initial potential, scan rate and accumulation time were optimised. Optimum conditions for the DPASV technique was obtained as follows: initial potential E_i = -1600 mV vs. Ag/AgCl (satd.); scan rate ρ = 2 mV per sec.; pH = 5.0; deposition time of 10 sec. Based on the DPASV of cadmium ion peak height at -0.78 V vs. Ag/AgCl (Sat'd), the c-MWCNT was found to enhance the anodic peak current of cadmium ion by a factor of 7 fold compared to that peak produced using a bare GRC electrode. Linear calibration curves were obtained from 1 ppb to 5 ppb with detection limit of 0.004 ppb and limit quantification of 0.012 ppb (R^2=0.966) respectively. The results suggest that the newly developed c-MWCNT/GRC has a potential to be a simple, efficient, low cost and disposable electrode system for the determination of cadmium ions at a very low concentration level.

ABSTRAK

Penentuan ion kadmium pada kadar surih dan sub-surih masih mencabar kerana kos analisis yang tinggi dan keupayaan instrumentasi yang terhad. Satu kaedah yang mudah, berkos rendah, menggunakan grafit tetulang karbon (GRC) elektrod diubahsuai dengan karbosilik tiubnano karbon multi berdinding (c-MWCNT) telah disediakan melalui kaedah elektroenapan dan digunakan untuk penentuan ion kadmium pada kadar sub-per bilion (sub-ppb). Kajian ini melibatkan penentuan prestasi elektrokimia GRC pada kekerasan dan saiz yang berbeza. Pencirian karbosilat MWCNT yang difungsikan adalah menggunakan kaedah Spektroskopi Inframerah Fourier Transformasi (FTIR) dan Bidang Pelepasan Imbasan Mikroskop Elektron-Tenaga Sebaran sinar-X (FESEM-EDX). FESEM juga digunakan untuk menyiasat morfologi permukaan elektrod c-MWCNT/GRC. Elektrod yang baru dibangunkan ini telah berjaya digunakan untuk pengesanan ion kadmium dalam penimbal 0.04 M Britain Robinson (BRB) melalui kaedah voltammetri perlucutan anodik denyut pembeza (DPASV). Beberapa parameter penting bagi operasi ini termasuk pH larutan penimbal, potensi awal, kadar imbasan dan masa pengumpulan telah dioptimumkan. Keadaan optimum yang dicapai untuk teknik DPASV telah diperolehi seperti berikut: potensi awal Ei = -1600 mV vs Ag / AgCl; kadar imbasan v = 2 mV per saat; pH = 5.0; masa pemendapan pada 10 saat. Berdasarkan voltamogram DPASV yang diperolehidengan ketinggian puncak ion kadmium pada -0.78 V vs Ag / AgCl, kehadiran c-MWCNT telah didapati dapat meningkatkan puncak anodik ion kadmium pada faktor 7 kali ganda berbanding puncak yang dihasilkan menggunakan elektrod GRC tidak terubah suai. Julat keluk penentu ukuran untuk teknik DPASV diperolehi daripada 1 ppb hingga 5 ppb dengan had pengesanan 0.004 ppb dan had kuantifikasi pada 0.012 ppb (R2 = 0.966). Keputusan menunjukkan bahawa elektrod c-MWCNT/GRC ini mempunyai potensi untuk menjadi satu sistem yang mudah, cekap, berkos rendah dan boleh dipakai buang bagi tujuan penentuan ion kadmium pada tahap kepekatan yang sangat rendah.